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Transport properties improvement and grain enlargement of $YBa_2Cu_3O_{7-\delta}$ superconductor by control annealing on Au foil

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Abstract . We have described a new process to prepare the aligned $YBa_2Cu_3O_{7-\delta}$ (YBCO) superconducting sample by unique annealing process on a gold foil. The transition temperature (T_c) of the Au doped samples is found to increase from 89 K to 94 K as the Au concentration is increased from 0 1 to 0 3% The current density (J_c) measured from M-H curve by Bean model for aligned pellet is ~ 2 × 10^4 Acm^{-2} Elongated aligned grains of YBCO ~ 200 μm long can be seen in the vicinity of this compound.

Keywords : Superconductivity, aligned grains, magnetization

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1. Introduction

Due to the technical importance, several groups have made efforts to dope $YBa_2Cu_3O_{7-\delta}$ (YBCO), high temperature superconducting material in bulk form, with the noble metals Au and Ag [1-3]. In the case of Au, when a small amount of Au is added in YBCO, Cu I sites are replaced by Au ions and c-axis lattice parameters expand without changing the oxygenation or the orthorhombic crystal structure and resistive transition temperature (T_c) increases by 3K [2]. If more Au metal is added, precipitated Au are observed between the YBCO grains and the critical current densities (J_c) drop and weak link behaviour is observed in applied magnetic fields [1, 3]. Au doped YBCO films show weakened superconductivity and no evidence of any microstructural degradation in the grains or at the grain boundaries in the films [4]. In the case of platinum, the densification and alignment were observed when YBCO pellets were annealed on platinum foil at 1020°C and this led to increase in J_c and aligned grains of YBCO ~ 250 μm [5]. In the present work, we have observed both densification and significant alignment in pellets made from reacted YBCO powder after annealing in flowing oxygen on a gold foil.

2. Preparation

The preparation of the $YBa_2Cu_3O_{7-\delta}$ was carried out by mixing Y_2O_3, $BaCO_3$ and CuO in the correct stoichiometric ratio and reacting the mixture in air at 930°C with two intermediate grinding stages. The reacted powder was pressed into pellets (of diameter 10 mm and thickness 2 mm) using a pressure of 154.4 MPa. The pellets were then placed on a gold foil with dimensions 2.5 × 2.5 × 0.1 mm (99.995% purity from Johnson Matthey) and annealed in flowing oxygen. The pellets were heated to 650°C at 2°C min^{-1}, held at 650°C for 2h, heated to 950°C at 5°C min^{-1}, held at 950°C for 2h, heated to 1020°C at 5°C min^{-1}, held at 1020°C for 2h, cooled to 1010°C at 10°C min^{-1}, held at 1010°C for 1/2h, cooled to 950°C at 1°C min^{-1}, held at 950°C for 2h, cooled to 450°C at 1°C min^{-1}, held at 1020°C for 12h, and then slow furnace cooled to room temperature (smaple B). For a few samples, the final annealing temperature was increased to 1030°C at 5°C min^{-1}, held at 1030°C for 1h, cooled to 1020°C at 10°C min^{-1}, held at 1020°C for 2h (sample C). Other procedure the same like sample B. For comparison, we also study the parent pellets (sample A). For a few samples, the final annealing temperature was increased to 1030°C. A few pellets from the same batch of reacted powder were also annealed on an alumina sheet following the above procedure for the purpose of comparison.

3. Results and discussion

We consider first sample A just annealed in oxygen flow at 950°C (parent sample). Sample B, annealed at 1020°C for 2h on gold foil in oxygen flow and sample C, annealed at 1035°C (near

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melting point) for 2h on gold foil in oxygen flow. In X-ray powder diffractometer, we used CuKα radiation All the samples were found to form single phase materials and indexed exactly in the same manner as that in the parent orthorhombic YBCO compound. Except that the position of 001 peaks are shifted reflecting a change in the c lattice parameter. As shown by the earlier investigators [1,2], the 006 peak in the X-ray pattern shifts to lower 2θ values while the 020 and 200 peaks remain stationary as are shown in Figure 1.

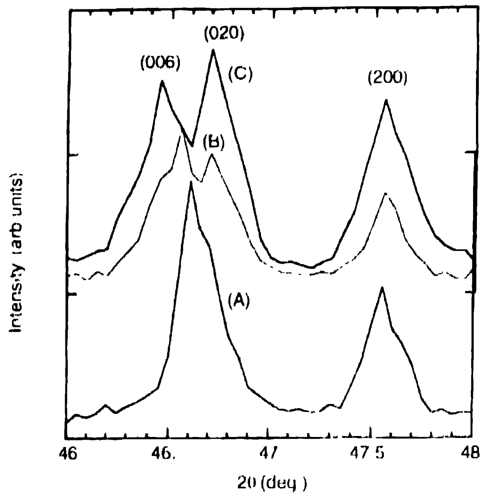

Figure 1. Part of the X ray diffraction pattern of samples (A) parent sample, (B) grain oriented sample and (C) densified sample Shows shift of 006 reflection as 020 and 200 remain constant

The melting behaviour of YBCO pellets during the annealing on the platinum [5] and gold foils is significantly different from that on alumina. The pellets on platinum and gold foils always retained their shapes in spite of being heated close to the melting temperature (~ 1035°C) while that on alumina decomposed and melted completely

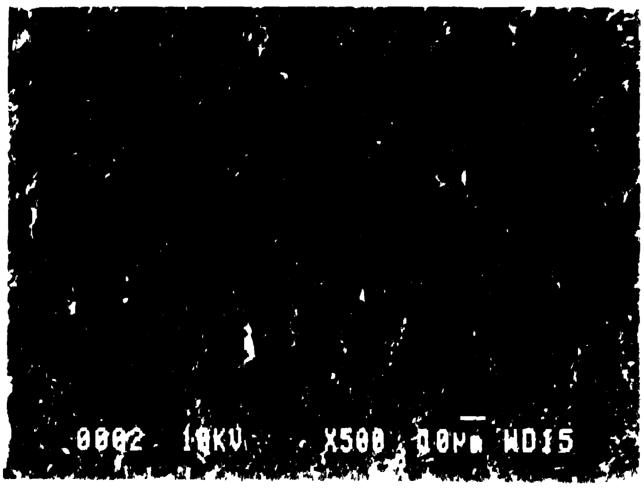

Figure 2. Scanning electron micrograph of the densified pellet annealed on gold foil at 1035°C for 1h

The elemental compositions of the samples were determined by the electron probe micro analysis (EPMA) revealing an atomic ratio of 1:2.3 for Y, Ba and Cu in the samples. The gold concentration of 0.5 to 1 at % in sample B and 2 to 3 at % in sample C was present and mainly confined to the grain boundaries. The scanning electron micrograph of the sample C is shown in Figure 2, where it can be noted that the sample is highly densified and the features that would be typical of the polycrystalline micro-structure of a sintered material are absent The density of the pellet was 93% of the theoretical density of YBCO. In the case of parent material, sample A has 76% and sample B shows 85% of the theoretical density of YBCO. At the temperature of 1020°C, the gold fills the grain boundaries and attracts the YBCO grains closer together and this is seen in the EDX investigation. In the vicinity of the gold near the interface, long (~ 200 μm) aligned grains of YBCO can be clearly seen in Figure 3. Due to this important nature of gold, gold crucible is most suitable for growing the big single crystal of YBCO.

Figure 3. Aligned grains of YBCO observed in samples, annealed on gold foil at 1020°C for 2h

Four-point resistivity measurements were made in a closed cycle cryostat. As shown in Figure 4, the superconducting transition temperature (T_c) increases with the increase in gold concentration. The dense sample C shows T_c (zero) ~ 94K higher than both, the grain oriented sample B, T_c (zero) ~ 92K and parent sample A, T_c (zero) ~ 89K. The same evidence was found by DC susceptibility measurement. The DC susceptibility as a function of temperature was measured using a DC magnetometer

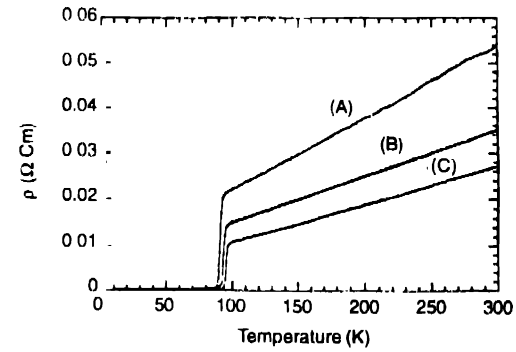

Figure 4. Resistivity vs temperature data of samples (A) parent sample (B) grain oriented sample and (C) highly densified sample. Shows T_c (zero) 98, 92 and 94K respectively

The sample was cooled in zero field and the susceptibility is shown in Figure 5, where a sharp superconducting transition at T_c ~ 94.4K is evident for dense sample C and sample A and B show T_c ~ 90K T_c ~ 92K respectively.

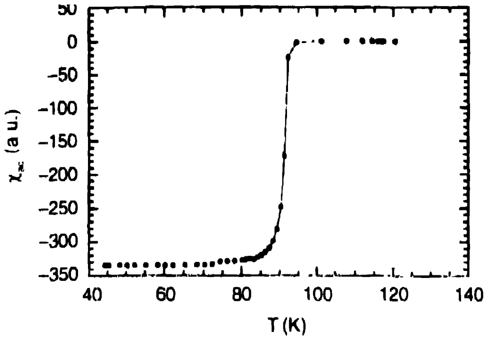

Figure 5. DC susceptibility of the densified sample, annealed on gold foil at 1035°C for 1h

The magnetization as a function of magnetic field intensity were measured using a SQUID magnetometer. A plot of magnetization as a function of a c-axis magnetic field is shown in Figure 6. The magnetization shows a sharp minimum located at 1.8 KG followed by an approach to saturation. The magnetization curve is similar to that obtained by other workers in single crystal YBCO [6] and we have calculated the J_c of these samples by Bean model [7] and found that the J_c increases from 4×10^3 to 2×10^4 A cm^{-2} towards going for densification samples.

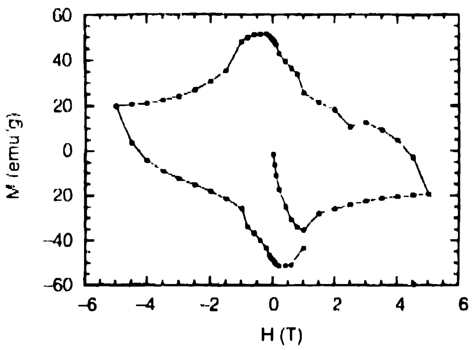

Figure 6. M vs H curve for the densified sample, annealed on gold foil at 1035°C for 1h

In this work, we have described a new process for the preparation of the gold dense YBCO superconductor. The increase of c-axis parameter of the YBCO unit cell is correlated with a systematic increase in the T_c value as measured in a transport experiment of the materials. Due to this process, the morphology of the samples is changed when annealed at 1020°C for 2h and the crystallites are oriented. The gold has increased the melting point of the YBCO and filled the grain boundaries of the YBCO, when we annealed at 1035°C for 1h. This caused increase in the densification as well as the critical current density of the materials.

4. Conclusions

We have discovered a novel and simple process of preparing the dense sample of YBCO. We have demonstrated the densification and alignment of YBCO pellets by annealing on a gold foil. The next step is to attempt to repeat the procedure for YBCO wires contained in gold tubes with the aim of improving the principal properties of the high T_c wires.

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